CHAPTER III

EXPERIMENTAL

3.1 Materials

3.1.1 Chemicals

All chemicals used in this work are shown in Table 3.1.

Table 3.1 Chemicals used in this work

Chemical	Source	Purity
Methanol	Aldrich	99.8 %
Ethanol	Aldrich	99.5 %
n-Propanol	Aldrich	99.9 %
n-Butanol	Aldrich	99.7 %
3-aminopropyl - trimethoxysilane	Aldrich	97.0 %

3.1.2 Adsorbents

Commercially available adsorbents were provided by UOP LLC. There were zeolites (silicalite and LZ-210), activated carbon (0.5 % Ash), polymeric resin (XAD-2), and silica gel (grade 636). Silica Hi-Sil®255 and modified silica Hi-Sil®255 were obtained from The Petroleum and Petrochemical College, Chulalongkorn University, Bangkok, Thailand.

In addition to the above adsorbent, silica gel was treated with different solvents to adjust its polarity. Chemicals used for the treatment were methanol, ethanol, n-propanol, n-butanol, and 3-aminopropyltrimethoxysilane. 40 mL of silica gel was dried at 110°C in an oven for 3 hours to remove surface adsorbed water molecules. The dried silica gel was suspended in 60 mL of a solvent, and then the mixture was put in an autoclave and heated at 100°C overnight. After that, the

product was filtered, washed with ethanol, and dried in an oven at 80°C for 3 hours to remove the unreacted solvent and stored in a dessicator until needed.

3.2 Experiments

3.2.1 Single Component Adsorption Experiment

3.2.1.1 Moisture Adsorption Experiment

Crucibles were heated at 400°C for 3 hours (for silicalite) and cooled to 120°C in a hot-air oven. They were moved and kept in a dessicator until used. The samples and the dried crucibles were weighed and then heated at 400°C for 3 hours. They were cooled to 120°C in an oven and then kept in a dessicator until they were cooled to room temperature. The dried samples were weighed and dried weights of the samples (W1) were calculated. The dried samples were placed in a closed humidification chamber for 24 hours and then weighed to determine the mass of water adsorbed (W2). The moisture adsorption capacity was calculated per unit mass of the sample (W2/W1).

3.1.2.2 Ethanol Adsorption Experiment

The same procedure described above was applied for the ethanol equilibrium adsorption experiments except the solution in a chamber. The dried samples were weighed and dried weights of the samples (W1) were calculated. The dried samples were placed in a closed chamber by saturating with ethanol for 24 hours and then weighed to determine the mass of the ethanol adsorbed (W2). The ethanol adsorption capacity was calculated per unit mass of the sample (W2/W1).

3.2.2 Competitive Component Adsorption Experiment

Equilibrium single component adsorption experiments of ethanol on adsorbents (silicalite, LZ-210, activated carbon, XAD-2, silica gel, and treated silica gel) were performed. Silicalite was calcined at 400°C for 3 hours. Solutions of ethanol-water mixtures were prepared with difference concentration of ethanol (1-12 wt%). Approximately, two grams of silicalite were put into a vial. Then, about 20 mL of the solution were injected into the silicalite-containing vial and a blank vial without silicalite. After the solution was injected, the vial was immediately sealed

and weighed. Samples were kept at room temperature (~ 25°C) and shaken frequently until they reached equilibrium. Liquid samples were taken and analyzed by a liquid chromatograph (LC) (Waters 2695 Separation Module) combined with a Waters 2414 Refractive Index detector at 40°C. The column was BioRad HPX-87H (300x7.8 mm) "ion-exclusion". The 0.0089N of H_2SO_4 was used as the mobile phase. Other adsorbents were conducted as the same procedure except the calcination temperature was 120°C for activated carbon, 400°C for LZ-210, and 90°C for XAD-2, silica gel, and treated silica gel.

3.2.3 Dynamic Adsorption: Breakthrough Curves

In this experiment, silicalite, activated carbon, LZ-210, XAD-2, silica gel, and silanized silica gel were used as the adsorbents and there was just only one composition of the feed. Figure 3.1 shows the experimental set-up for the dynamic unit. A dried adsorbent was packed in a 55 mL column with 0.78 and 114 cm as the inside diameter and length. Glass wood was placed at both ends of the column to block the adsorbent. The column was positioned in the controlled temperature hot box and connected to a fraction collector. Firstly, this experiment was conducted at 30°C. The experiment was started with feeding the solution, which was a mixture between 12% ethanol and 88% water, at a constant flow rate of 1.2 mL/min that continuously flowed through the column. At the same time, the fraction collector was started to collect the system effluent every 2 min. Each fraction, approximately, 2.4 mL of the sample, was analyzed by the liquid chromatograph (LC). After the column was saturated with the solution, a heater and fraction collector were turned off. The column was purged with nitrogen gas. Then, the temperature was increased to 40°C and 120°C, respectively for desorption. The effluents at 40°C and 120°C were analyzed by the liquid chromatograph (LC). The concentration of ethanol was plotted versus time or volume and then the ethanol adsorption capacity was calculated.

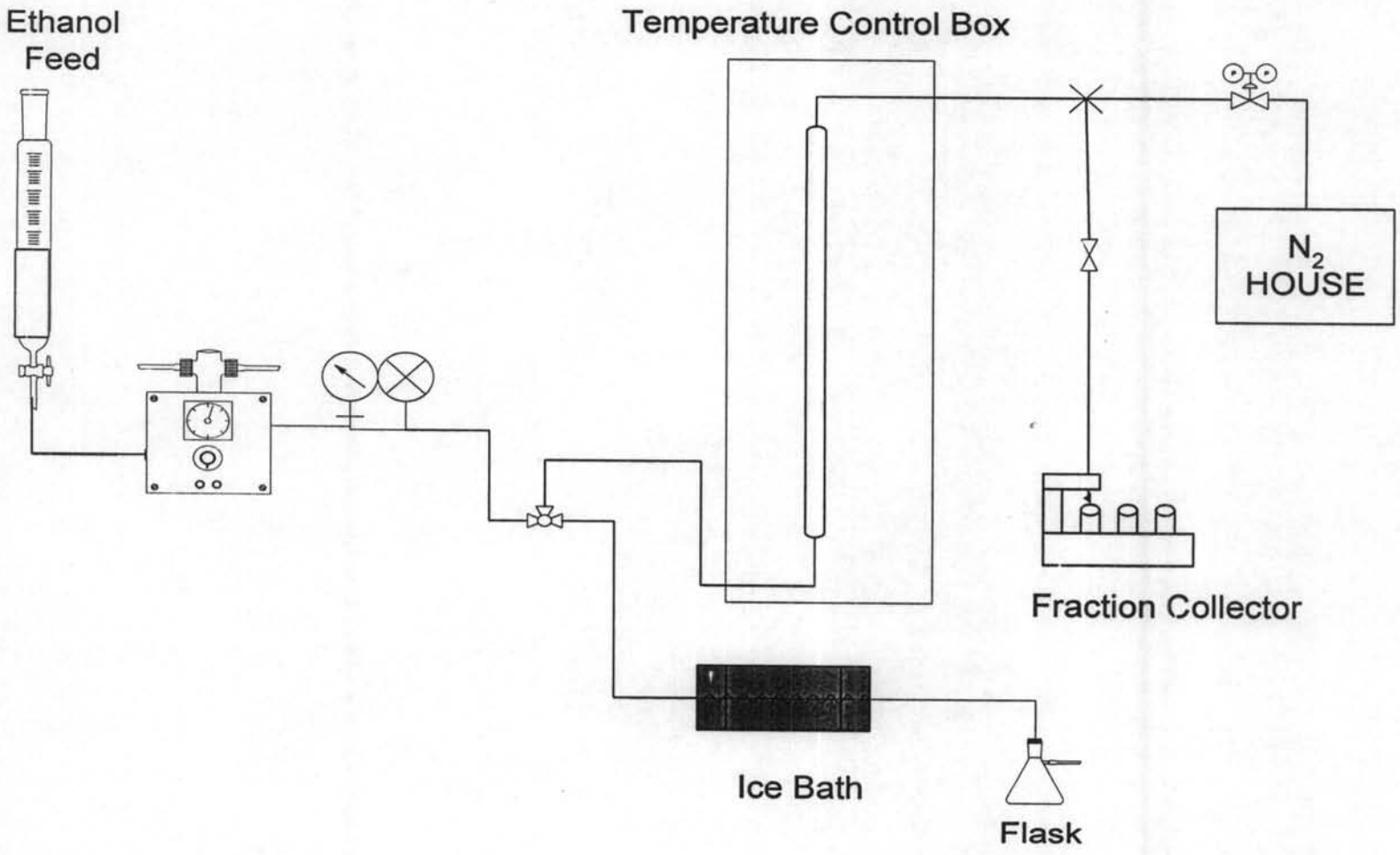

Figure 3.1 Experimental set-up of the dynamic unit.